

FINAL REPORT SUBMITTED TO  
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Aerospace and Materials Sciences Directorate  
High Temperature Materials Program

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Project Title:  
**MEANS 2: Microstructure- and Micromechanism-Sensitive Property Models for  
Advanced Turbine Disk and Blade Systems**  
**AFOSR Grant No: FA9550-05-1-0102**

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14. ABSTRACT  The overall coordinated research effort involved activities in the following areas: (i) Creep and Deformation Structure Characterization Studies of Disk Alloy Rene 104 (ME3), (ii) Calculation of Activation Barriers Using Phase Field Dislocation Dynamics, (iii) Adaptive computational model of crystal plasticity involving micro-twinning, (iv) 3D Atom Probe Analysis of Phase Microstructures. Descriptions of all activities can be found in the annual and final reports submitted and to be submitted by the overall PI, Mike Mills.					
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## I. EXECUTIVE SUMMARY

This effort comprised a coordinated team of researchers from the Ohio State University (under Grant #FA9550-05-1-0135), University of Michigan (supported by #FA9550-05-1-0100) and Johns Hopkins University (supported by #FA9550-05-1-0102) and was entitled, MEANS 2: Microstructure- and Micromechanism-Sensitive Property Models for Advanced Turbine Disk and Blade Systems. The overall MEANS-2 team focused on verification and refinement of understanding of mechanisms and their transitions at intermediate temperatures in the disk alloy Rene 104. Evidence of microtwinning, continuous faulting and dislocation by-pass has been observed at successively higher temperatures above 650°C. Modeling at the ab initio, atomistic and phase field levels is providing important insight into the activation parameters associated with the observed deformation mechanisms at OSU; a microtwinning model has been found to provide reasonable agreement with the experimental creep response for Rene 104 and Rene 88. A novel phase field model of directional coarsening (rafting) during high temperature, low stress creep of blade alloys has also been developed; this model accounts for the local stress fields associated with matrix dislocations as well as the lattice misfit, and demonstrates promising qualitative agreement with experiment. Single crystals of Rene 104, a polycrystalline disk alloy, have successfully been grown at the U of M. These crystals have been heat treated to produce an appropriate  $\gamma$ - $\gamma'$  microstructure and single-crystalline micro-tensile/compression specimens have been oriented and prepared at JHU. Micro-scale experiments to allow for measurement of the orientation and tension/compression creep response of Rene 104 have also been developed at JHU. As a result of these efforts, we are poised to measure creep anisotropy and tension-compression asymmetry as a function of crystal orientation and to provide data that will assist in the development of crystal plasticity models for this alloy.

## II. RESEARCH OBJECTIVES

The goal of the program was to develop improved models that: (a) incorporate more realistic representation of the relevant microstructures and micromechanisms, (b) enable modeling for a range of relevant service conditions, (c) address time-dependent deformation in both disk and blade alloys, (d) investigate crack initiation in blade materials, and (d) provide this information even more accessibly to the component design process, building upon the paths to the design process created in the DARPA Accelerated Insertion of Materials (AIM) program.

Tresa Pollock (PI at UM) led efforts to characterize blade alloy microstructures and provided single crystals materials for the rest of the program. Kevin Hemker (PI at JHU) developed micro-tensile and compression tests for single crystal variants of the disk alloy. The work focused on a next generation Ni base disk alloy (Rene 104) and a turbine blade alloy (Rene N5) which are of keen, common interest to our industrial collaborators, GEAE and P&W, for both military and commercial propulsion systems.

### III. ACCOMPLISHMENTS/ NEW FINDINGS

The overall coordinated research effort involved activities in the following areas: (i) Creep and Deformation Structure Characterization Studies of Disk Alloy Rene 104 (ME3), (ii) Calculation of Activation Barriers Using Phase Field Dislocation Dynamics, (iii) Adaptive computational model of crystal plasticity involving micro-twinning, (iv) 3D Atom Probe Analysis of Phase Composition, Ordering and Interface Profiles, and (vi) Deformation Mechanisms for Rafted Microstructures. Descriptions of all activities can be found in the annual and final reports submitted and to be submitted by the overall PI, Mike Mills. Activities at JHU focused on the first task, Creep and Deformation Structure Characterization Studies of Disk Alloy Rene 104 (ME3), which is the focus of this report.

The overarching objective of this task was to enhance the fidelity of the elevated temperature creep mechanism map that has been developed by Mills and colleagues to describe the creep behavior of Rene 104, see Fig. 1. Activities include: identifying transitions and additional regimes, conducting transient creep tests to determine activation energies and stress dependencies, examining creep anisotropy and tension-compression asymmetry to determine the anisotropy of creep/plasticity with orientation for crystal plasticity models.

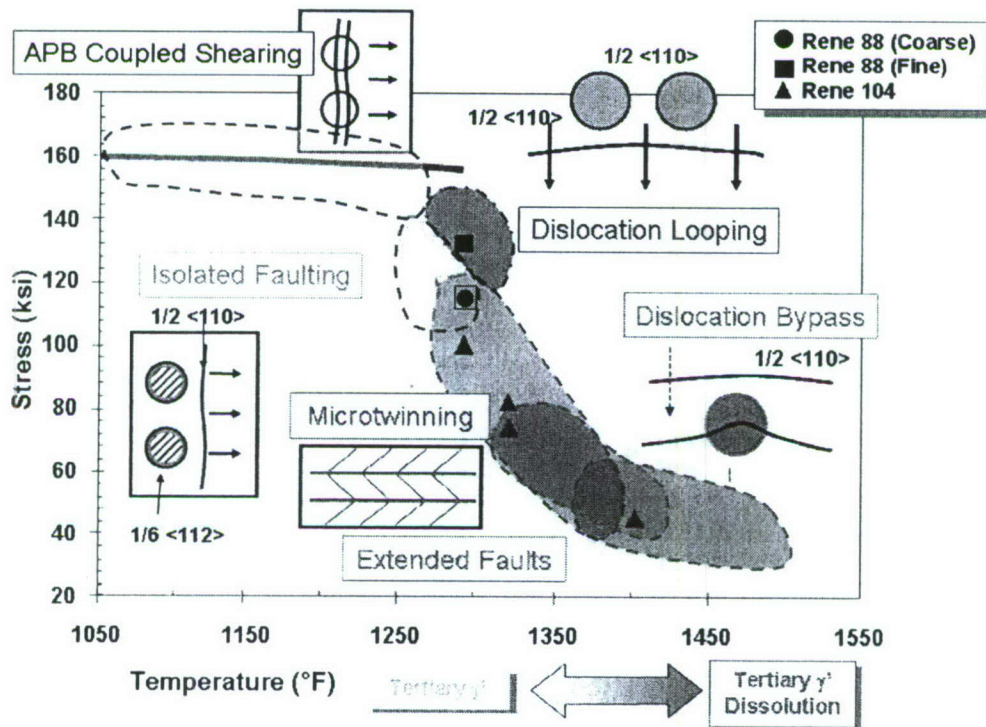
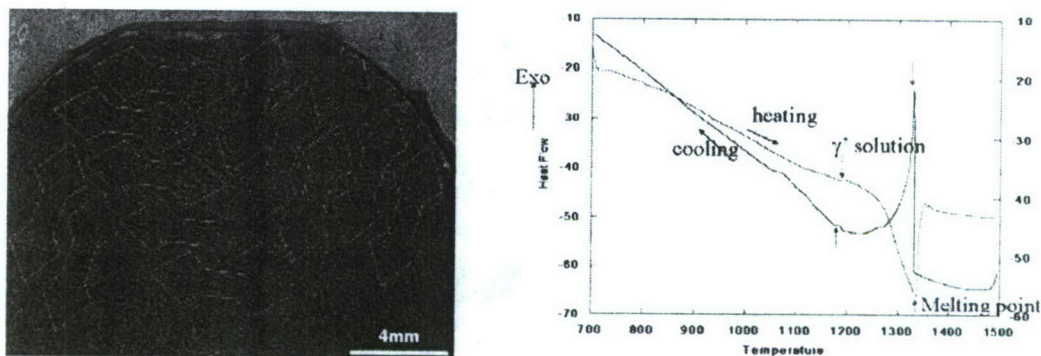


Fig. 1: Mechanisms identified for creep of ME3 over a range of stresses and temperatures, with approximate transitions in mechanisms indicated (after Mills).



Rene 104 is a polycrystalline alloy, but measurements of the orientation dependence of creep anisotropy and tension-compression asymmetry, that are needed as input for crystal plasticity models, can only be made using single crystalline specimens. Initial attempts to grow Rene 104 single crystals in the Crystallox at the University of Michigan (UM) failed to produce high quality crystals, and considerable time was required to grow crystals from seeds using a four bar mold technique. Crystals that are 1-inch in diameter and 6-inches in length were grown in the second year of the study and the third year was spent preparing those crystals for testing. Slices 700-800 $\mu$ m thick were cut perpendicular to longitudinal axis of each rod using a wire EDM, polished to a mirror finish, and etched to reveal grain boundaries. From this procedure it was determined that although not perfectly single crystalline, the grains present in these rods were of significant size such that single crystalline micro-tensile samples could be prepared from within individual grains (fig 2a).



**Fig. 2:** (a) an etched cross-section of single crystal ME-3 grown at UM demonstrating significantly large grain size. Micro-tensile and compression specimens are overlaid to illustrate the relative size and orientation of the specimens to be made. (b) DTA of this crystal showing a melting point of 1330°C and  $\gamma'$  solvus temperature of 1183°C.

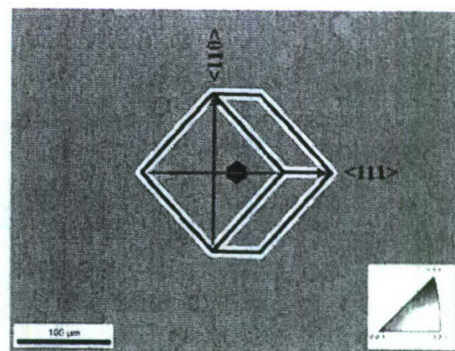
The chemical composition of the rods was confirmed and differential thermal analysis (DTA) used to determine the melting point and the  $\gamma'$  solution temperature of the crystals (fig 2b). These measurements were used to determine the heat treatments needed to produce a realistic gamma-gamma prime microstructure. With help from our colleagues at OSU we homogenized the crystal at 1250°C for 23 hours, cooled and sliced into pieces. Each piece was then heated above the solvus temperature of 1183°C and held for 1 hour, before being cooled at different rates, thus producing a fine (fast cooled) and coarse (slow cooled)  $\gamma$ - $\gamma'$  microstructure.

Orienting these samples using the back reflection Laue technique proved more difficult than originally expected. After multiple attempts to orient the crystals at JHU and OSU were unsuccessful, attention instead turned to electron backscattered diffraction (EBSD) orientation imaging microscopy (OIM) which was successfully implemented and used to orient significantly large grains of both the slow and fast cooled microstructures. Numerous grains of specific orientations have been identified, sliced, polished and prepared as micro-tensile/compression specimens. Fig. 3 shows an OIM pattern of a slice that was cut from an oriented crystal. The slice contains the {112} plane with the  $\langle 110 \rangle$  and  $\langle 1\bar{1}1 \rangle$  directions oriented as shown. Micro-specimens have been prepared along  $\langle 110 \rangle$ ,  $\langle 1\bar{1}1 \rangle$  and 45° from each



direction such that the maximum shear stress of that tensile / compressive axis aligns with both directions.

The development of micro-tensile testing techniques by Sharpe and subsequent extensions to elevated temperature testing by Hemker has greatly facilitated the mechanical testing of small specimens at JHU. The micro-testing laboratory contains a suite of instruments especially designed to measure the mechanical response of micron-sized specimens over a range of temperatures (25-1500°C), strain rates ( $10^{-2}$ - $10^{-8}$  s<sup>-1</sup>), and loading conditions (constant displacement, constant load, micro-bending, stress relaxation and cyclic fatigue). The workhorse of this laboratory is a micro-sample tensile machine with: self-aligning grips, a linear air bearing, piezo-actuator, miniature in-line load cell, environmental chamber, resistive heating, interferometric strain/displacement gage (ISDG), and a digital image correlation (DIC) system. Hemker has used free-standing micro-samples to measure the mechanical response of a wide range of bulk metals and alloys, nickel aluminide bond coats, and coatings for rocket motor liners. In the current project, the test system was modified to allow for extended constant load creep testing in both tension and compression. The diamond shaped shoulders shown in Fig. 2a facilitate self-alignment in both directions, and the use of real-time video capture and digital image correlation allow for localized strain measurement.



**Fig. 3:** OIM pattern of a slice of ME-3 crystal used to orient and prepare micro-specimens.

Overcoming the obstacles associated with the preparation of single crystalline micro-sample and the development and implementation of micro-tensile/compressive creep testing has taken three years. We have requested a 6-month extension allow us to complete these experiments and Tables 1 and 2 present a plan for the single-crystalline micro-sample creep experiments to be conducted. The result of that study will be delivered to our OSU colleagues and used to compliment their micro-mechanical models. The merits of the current study lie in the preparation of single crystalline specimens and unique test techniques for those experiments.

**Table 1: Constant strain-rate tension/compression experiments to be conducted.**

Plane	Direction	Microstructure	Temperatures
{112}	<110>	Fine/Coarse	RT, 600°C-900°C
{112}	<111>	Fine/Coarse	RT, 600°C-900°C
{112}	45° from <110> , <111>	Fine/Coarse	RT, 600°C-900°C
{110}	<001>	Fine/Coarse	RT, 600°C-900°C

<b>Table 2: Creep tension/compression experiments to be conducted.</b>			
<b>Plane</b>	<b>Direction</b>	<b>Microstructure</b>	<b>Temperatures</b>
{112}	<110>	Fine/Coarse	600°C-900°C
{112}	<111>	Fine/Coarse	600°C-900°C
{112}	45° from <110> , <111>	Fine/Coarse	600°C-900°C
{110}	<001>	Fine/Coarse	600°C-900°C

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#### IV. JHU PERSONNEL SUPPORT ON THIS GRANT

- Professor Kevin J. Hemker; PI;  
*AFOSR supports 1 month salary per year*
- Dr. Piyush Jain; Ph.D. graduate student.  
*Received Ph.D, May 2007.*
- Mr. Daniel Butler; PhD. graduate student.  
*Ph.D. expected September 2008.*

#### V. PUBLICATIONS

- None yet.

#### VI. TRANSITIONS

- *Specific to JHU:* Dan Butler visited the General Electric Global Research Center in Niskayuna, NY, in October 2007 and delivered a seminar entitled, "Tension-compression asymmetry and orientation anisotropy of a Ni-based turbine disk alloy".
- *Overall:* Extensive discussions and exchange of information has already occurred with our industrial partners at GE Aircraft Engines (Dave Mourer, Mike Henry and Deb Whitis) and Pratt-Whitney (Michael Savage). Characterization and modeling of creep mechanisms are serving as a basis for understanding low cycle fatigue in similar disk alloys in the NASA Propulsion 21 program.

## NEW DISCOVERIES, INVENTIONS, PATENT DISCLOSURES

- None

## VII. HONORS / AWARDS

- Hemker was elected Fellow of American Society of Mechanical Engineering (ASME), December 2007.